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## **Project Summary**

## Sediment Sampling Quality Assurance User's Guide

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This report is to serve as a companion to an analogous document on soil sampling quality assurance. Prior to the design of an adequate quality assurance/quality control (QA/QC) plan for sediment sampling, there must be agreement on the objectives of the sampling program. Answers to the following questions should be available: How will the resulting data be used to draw conclusions? What actions may be taken as a result of those conclusions? What are the allowable errors in the results? Once answers to these questions are available, an experimental protocol may be prepared with an appropriate statistical design and QA/QC plan.

An overview of selected sediment models is presented to serve as a foundation for stratification of study regions and selection of locations for sampling sites, methods of sampling, and sample preparation and analyses. Discussions of situations relating to rivers, lakes, and estuaries are included.

Statistical considerations presented include experimental statistical designs to enable ANOVA to be accomplished, discussion of Type I and Type II errors, numbers and locations of sampling sites, bias, confidence and prediction limits, outliers, and testing hypotheses. The importance of an exploratory study to the cost-effective achievement of the overall objectives of a sediment sampling program is emphasized.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Las Vegas, NV, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

## Introduction

U.S. Environmental Protection Agency (USEPA) quality assurance policy requires that every monitoring and measurement project must have a written and approved quality assurance (QA) project plan. Among the sixteen elements which must be contained in all QA project plans are the following:

- Project description.
- QA objectives for measurement data in terms of precision, accuracy, completeness, representativeness, and comparability.
- Data analysis, validation, and reporting.
- Specific routine procedures used to assess data precision, accuracy, and completeness.

This report, which is a companion to an analogous document on soil sampling quality assurance, addresses selected factors associated with the application of quality assurance/quality control (QA/QC) guidelines to sediment sampling. In order to make this report more self-contained, chapters from the companion soil report covering such topics as sample handling, analysis of QA/QC data, and system audits, which are equally applicable to sediment sampling, are contained verbatim in the appendices.

The most important consideration for sediment sampling is the objective for which the sampling is being done. The statement of objectives should contain clear answers to the following questions:

- How will the resulting data be used to draw conclusions?
- What actions may be taken as a result of those conclusions?
- What are the allowable errors in the results?

Once answers to these questions are available an appropriate statistical design for the sampling and analysis program, to include an adequate and verifiable QA/QC project plan for the study, can be devised.

Prior to the establishment of an adequate, cost-effective QA/QC plan for sediment monitoring programs, a decision-making official, after careful analysis of the consequences, must specify allowable Type I and Type II errors in the results. A Type I error, for a situation in which a measured population mean is being compared to either an action level or a control level, is committed when it is concluded that the population mean exceeds the action or control level when in fact it does not. For the same situation, a Type II error is committed when it is concluded that the population mean does not exceed the action or control level when in fact it does. The desired minimum detectable difference between a measured population mean and either an action, or a control level must also be specified.

The goal of this document is to provide a flexible, but technically sound, framework within which the user can devise a QA/QC plan consistent with the specific objectives of any sediment monitoring program. The document has been developed to serve as a user's guide for anyone designing, implementing, or overseeing sediment monitoring programs.

The extent to which adequate field-validated models exist for describing sediment transport and deposition has a direct bearing on the design of cost-effective sediment monitoring programs. Generally, when adequate models exist, fewer monitoring measurements are required to assess pollutant levels and their significance. Accordingly, this report presents a brief review of some available sediment transport models after first providing some background definitions and discussions.

The models range from simple, steady state, dissolved oxygen relationships to very complex models describing the interrelationships among pollutant additions and removals, organic matter concentrations, and life processes occurring in

aquatic environments. Many pollutants can be transported in suspended solid form or adsorbed on suspended particulates. Unfortunately, the dynamics of the movement of pollutants adsorbed on sediments is not well understood.

Sediments play an important role in the transport of pollutants as well as in the transport of nutrients. Both the pollution and nutrient aspects must be considered. Sediments can overwhelm bottom fauna, but the nutrients they carry can give rise to new biota.

In choosing an appropriate model, a comparison should be made of available models. A model should be fitted to the problem and not vice versa. If complete validated models are not available for the pollutants and other site-specific conditions of a problem, it still may be possible to use portions of available models, or other empirical field experience in the cost-effective design of sediment sampling programs.

The responsibilities of National Program Managers in the USEPA Mandatory Quality Assurance Program include ensuring that data quality acceptance criteria and QA Project Plans are prepared for all data collection projects sponsored by their offices.

This requires the development of data quality objectives (DQOs). DQOs are qualitative and quantitative statements developed by data users to specify the quality of data needed from a particular data collection activity.

DQOs are the basis for specifying the quality assurance and quality control activities associated with the data collection process. QA Project Plans clearly describe what will be done at each stage of data collection (i.e., sample site selection, sample collection, sample handling and analysis, and data handling and analysis) and include instructions or standard operating procedures for each field and laboratory activity.

Some possible objectives for sediment sampling are:

- Determining the extent to which sediments act as either sources or sinks for water pollutants,
- Determining presence and distribution of selected pollutants in sediments in both space and time,
- Determining the risk to human health and/or the environment from sediment contamination by selected pollutants, and
- Taking measurements for validation of sediment transport and deposition models.

Under most circumstances, background data will not be available for a given monitoring location. These data must be acquired before, or preferably during, any sediment monitoring program. The intensity of the background sampling that is undertaken depends upon the pollutants being measured, the sediment characteristics and variability, the levels of pollutant likely to be found in the study area and the purpose of the study. QA/QC procedures are just as critical for the background measurements as they are for the study area measurements.

When sediments are contaminated, drinking water or human foods, contaminated directly or indirectly through contact with sediments, may be unfit for human consumption. As the hazardous constituents move through different trophic levels, substantial biomagnification of contaminants may take place.

The steps outlined below are designed to provide a sediment monitoring effort with minimal needed sample precision and representativeness.

- Determine the components of variance that should be built into the statistical design.
- Choose the allowable probabilities for Type I and Type II errors and the difference in means considered to be significant. (These are the DQOs and they are needed together with an estimate of the coefficient of variation to determine the number of samples required in each stratified region.)
- Obtain sampling data from studies with similar characteristics to the one of interest. (Estimates of coefficients of variation are of particular importance.)
- Calculate the mean and note the range of each set of duplicates (co-located independent samples).
- Using results from previous studies, develop a table of critical difference values for duplicate sample results for various concentrations that span the range of concentrations of interest. Use this table to accept or reject sets of duplicates.

Suggestions for additional elements of a more complete QA/QC plan are provided in the text.

The DQO guidelines below are suggested for the indicated operational situations.

	Confidence Level (1-α)	Power Relative (1-β) Increase*
Preliminary Site	-	
Investigation	70-80%	90-95% 10-20%
Emergency		
Cleanup	80-90%	90-95% 10-20%
Planned		
Removal and		
Remedial		
Response		
Activities	90-95%	90-95% 10-20%

<sup>\*</sup>Relative Increase from Background or an Action Level to be Detectable with Probability (1-\(\rho\)).

Statistical sampling plans are based on assumptions concerning the probability distributions of the measurements to be made. The properties of a normal distribution are so desirable that, if the data are not normally distributed, a transformation is sought to convert the existing distribution into a new distribution which is approximately normal.

The maximum probability allowed for a Type I error is called the significance level of the test of hypothesis and is commonly denoted by alpha ( $\alpha$ ). The probability of a Type II error is usually denoted by beta  $(\beta)$ and is typically a function of  $\alpha$ , sample size, and the size of the deviation from the null hypothesis. The probability that the alternative hypothesis will be accepted when it is true is called the power of the test and may be denoted by  $(1-\beta)$ . Typically, the experimenter will specify the smallest deviation from the null hypothesis that he considers to be scientifically, economically, or environmentally important to detect and then specifies the power of the test that he wants for that specific alternative.

The Quality Assurance Officer, supported by a qualified statistician, should be intimately involved in the review of the experimental or sampling design proposed by the investigator. He should insure that the information obtained provides measures of the components of variance that are identified in the field.

Composite samples provide only an estimate of the mean of the population from which the samples forming the composite are drawn. No estimate of the variance of the mean, and hence, the precision with which the mean is estimated can be obtained from a composite of samples. Since the primary purpose of QA/QC is to measure the precision of the samples obtained, the compositing of

samples should be avoided if at all possible.

Split samples, spiked samples and blanks are used to provide a measure of the internal consistency of the samples and to provide an estimate of the components of variance and the bias in the analytical process. The number of QA/QC samples needed is suggested as one out of every twenty samples for most categories of samples. In some instances this guideline may not be adequate while in others it may provide more samples than are necessary. It is good practice to perform an initial exploratory study in which, among other things, QA/QC samples in excess of the guideline recommendations are collected and analyzed. Analysis of the resulting data will provide a better estimate of the optimum required number of QA/QC samples of different types.

Typically, one wishes to estimate the concentration of measured pollutants in the sediments and to indicate the precision of these estimates. To indicate precision of an estimate, one may provide the standard error or a confidence interval for the expected value of the concentration. The confidence interval is bounded by confidence limits. Confidence limits are bounds of uncertainty about the average caused by the variability of the experiment.

Prediction limits are similar to confidence limits but are used to identify an interval into which a randomly chosen future sample value should fall. Equations for both confidence and prediction limits are provided along with an example calculation.

A problem that is particularly prevalent in data obtained from field samples is that of outliers. The cause of the outlier may be an error of procedure in sampling, subsampling, chemical analysis, or the transcribing of data; or it may be due to an anomaly that would indicate that a change is required in the assumed model for the process. Guidelines are provided for rejecting outliers, however, there are many problems with outlier tests. If at all possible, prior to rejecting values as outliers, repeat measurements should be made on the same or nearly identical samples.

Once objectives have been defined which involve the need for sediment sampling, the next step is to develop a total study protocol including an appropriate QA/QC project plan. The recommended approach is to conduct an exploratory study first that includes both a

literature and information search along with selected field measurements made on the basis of some assumed transport model.

To provide a framework for the discussion, a hypothetical situation involving an abandoned hazardous waste site is described. The established objective for this hypothetical situation is to conduct an environmental assessment of the site and its environs to determine whether a short or long term hazard to man or the environment exists. If a hazard exists, its nature and extent must be defined and appropriate recommendations made to bring the hazard under control. A study team is organized to address the problem and the sediment study group's task is to identify and make an assessment of potential problems associated with sediments in a nearby river and estuary.

Questions which must be answered, at least in part, by the exploratory study include:

- What wastes have been placed at the disposal site over what time periods?
- What chemicals in what amounts have escaped from the site via what transport routes and what is the present geographical extent of these chemicals?
- What adverse effects on human health or the environment have been reported in the site vicinity?
- What is an appropriate background region to use for the study?

Before taking any field measurements, a comprehensive literature and information search should be conducted to determine what information may already be available. The results of the exploratory study will provide information and field data that will serve as the basis for the design of a more definitive monitoring study. Thus, any field measurements taken should include appropriate QA/QC measures to determine the quality of the data.

The hypothetical case study is developed step by step. Data quality objectives are identified, a grid system is defined, the study area is stratified, a background region is selected, number and locations of sites for sampling are determined, and an appropriate QA/QC project plan is prepared.

In general, the simplest sampling tool deemed to be adequate should be used. The advantages and disadvantages of some bottom samplers and some coring devices are presented in tables.

One of the possibilities for error during the sampling process is discarding non-sediment material collected with the sediment samples prior to analysis. It is suggested that all such discarded material be retained. Ten percent of these samples should be sent to the analytical laboratory for analysis with the remainder being archived.

If the exploratory study is conducted well, it will provide some data for achieving the objectives of the study; it will provide data concerning the feasibility and efficacy of most aspects of the study design including the QA/QC plan; it will serve as a training vehicle for all participants; and it will pinpoint where additional measurements need to be made.

Following analysis and interpretation of the information and data resulting from the exploratory study, the next step is the design of the final definitive study. Any problems with the QA/QC plan noted should be solved by appropriate modifications of the plan. The procedure is illustrated by extending the hypothetical case study based on assumed data obtained from the exploratory study.

In view of conclusions reached on the basis of the assumed data, the following questions which should be answered in the definitive study are identified:

- How far down the stream are the sediments significantly contaminated?
- What are the relative contributions of surface water and groundwater to the contamination of sediments?
- How are the sediment levels changing as a function to time?
- What levels of contamination in human foods are derived directly or indirectly through contact with sediment?
- What is the impact of contaminated sediments on aquatic biota?
- How should the study area be stratified in the definitive study?

A table is provided giving the number of samples required in a one-side, one-sample t-test to achieve a minimum detectable relative difference at confidence level  $(1-\alpha)$  and power  $(1-\beta)$ . In this table the coefficient of variation varies from 10 to 35%, the power from 80 to 95%, the confidence level from 80 to 99%, and the minimum detectable relative difference from 5 to 40%. An equation is provided to calculate values not included in the table.

The required frequency of sampling depends on the objectives of the study, the sources and sinks of pollution, the pollutant(s) of concern, transport rates, and disappearance rates. Assessment of trends in time will establish whether sediment concentrations are increasing, decreasing, or remaining fairly level. Evaluations of these trends will be important to selection of appropriate remedial response measures.

The analysis and interpretation of QA/QC from the more definitive study

should show how all aspects of the total QA/QC plan combine to give an overall level of reliability for various aspects of the resulting data. Another goal may be to determine whether all QA/QC procedures used were necessary and adequate. It is desirable to provide summarized tables of validated QA/QC data in the final report. From such tables it is possible to determine bias; precision; component random errors associated with reproducibility, extract matrix, sample matrix, and sample homogeneity; interlaboratory precision; and uncertainty. Presentation of QA/QC data also contributes to the building of a body of data in the literature which allows comparisons to be made between and among studies.

Data from the more definitive study describing variations in sediment concentrations with depth will show how effective dredging to different depths might be in the removal of the contamination. If dredging is even contemplated, safe and effective methods for disposing of the dredge spoil must be available.

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The complete report, entitled "Sediment Sampling Quality Assurance User's Guide," (Order No. PB 85-233 542/AS; Cost: \$14.50, subject to change) will be available only from:

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